january, 1961

nlgi spokesman

journal of the national lubricating grease institute

Oxidation of Lubricating Greases

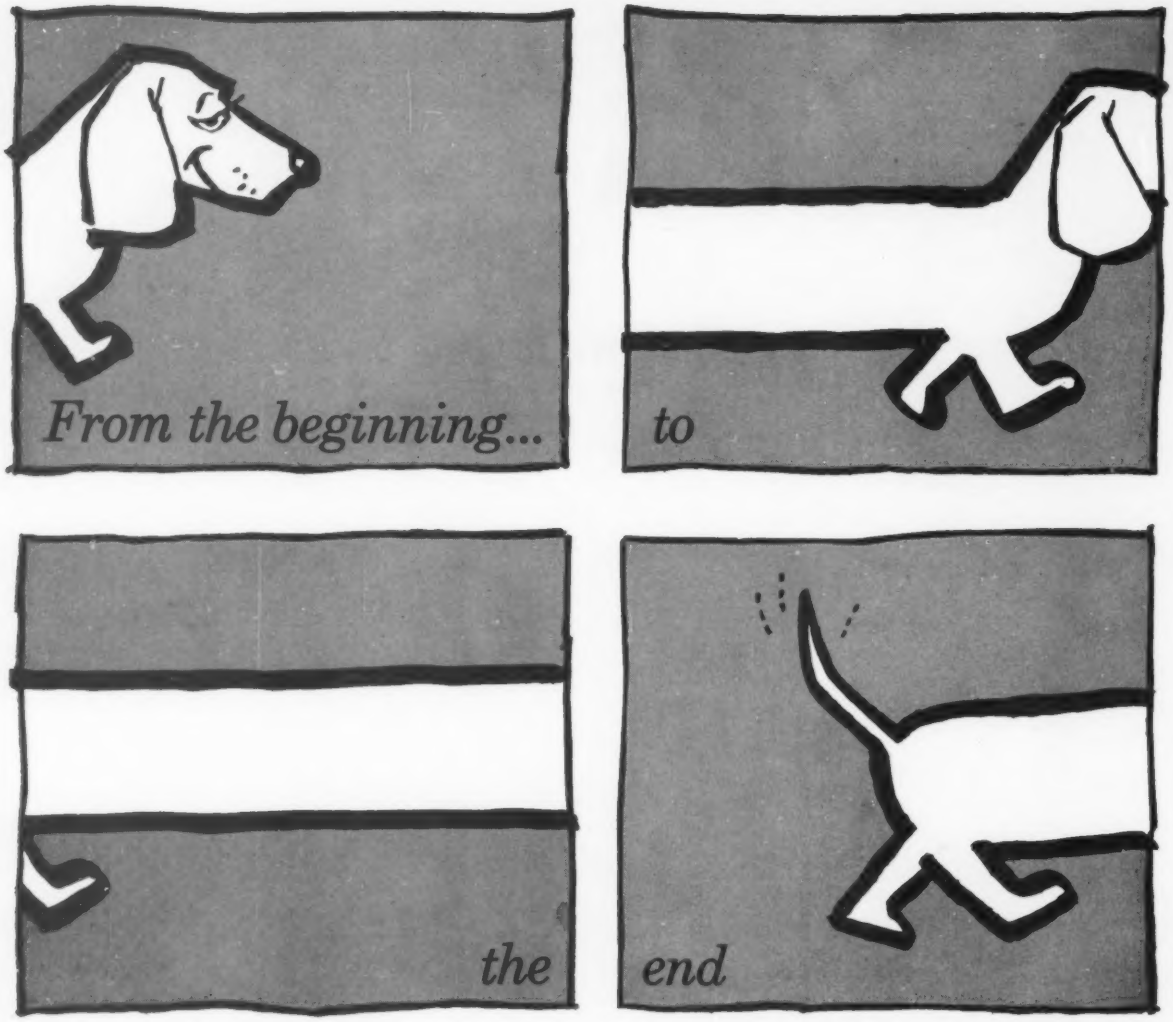
By J. E. GOODRICH and J. J. BURKE

The 1958 Food Additives Amendment—Its Effect on the Lubrication of Food Packaging Machinery

By A. E. DOOLEY

A Co-op First—Rapeseed Oil Grease

By M. A. McNICHOL



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Volume XXIV January, 1961 Number 10

Published Monthly by the National Lubricating Grease Institute, T. W. H. MILLER, Editor; VIRGINIA ALLEN, Assistant Editor, 4638 J. C. Nichols Parkway, Kansas City 12, Missouri. Telephone VAlentine 1-6771. 1 Year Subscription, $5.00. 1 Year Subscription (Foreign), $6.00.

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THE COVER

THERE is nothing about this picture to show that these men are working, but George Entwistle of Sinclair and L. C. Brunstrum from Standard of Indiana are doing their NLGI chores. Entwistle, as chairman of the important editorial review subcommittee, and Brunstrum, as chairman of the technical committee, devote long hours to industry affairs through the auspices of NLGI . . . this short session at a busy annual meeting is one way progress is implemented, by contributions in time and knowledge. The men elected and appointed to lead in NLGI activities are honored in this issue, beginning on page 394.

The NLGI SPOKESMAN is indexed by Industrial Arts Index and Chemical Abstracts. Microfilm copies are available through University Microfilm, Ann Arbor, Mich.

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NLGI PRESIDENT'S PAGE

By F. R. HART, *President*

What Does the Greaseless Chassis Bearing Portend?

Ever since the first model rolled off the assembly line, the design of American passenger cars has been changing and for the better. The more important changes come about slowly and are adopted only after the introductory prototypes prove successful. And so it has been with the design of the chassis. For example, it was only a few years ago that two wheel brakes were standard equipment and the front-end steering system consisted of a tie-rod assembly, steering arms, king pins and the other linkage that were used before the ball joint suspension arrived. Even the easy riding elliptical springs have been discarded in favor of coil springs and the torsion bar. Over the years, these changes were accepted without realizing that slowly we were losing a market for the 1,000-mile chassis lubrication job. Each chassis improvement eliminated moving parts and the grease fittings needed to keep them quiet. This loss will ultimately be felt by all manufacturers of lubricating grease but especially by the service station operator who depends on "grease jobs" as a source of revenue.

The situation may not be as hopeless as it sounds since the greaseless bearing, like the air-cushioned ride, may not be universally adopted; also, it could be discarded if the new design fails to lower cost. As far as the car manufacturer is concerned, each design change is intended to cut production costs, to cope with rising overhead and to meet competition. Most car manufacturers are primarily concerned with solving these problems, sometimes to the consternation of allied industry such as ours. We, the grease producer, find ourselves in the middle of this economic squeeze much the same as the ice-man found himself in the late 1920's. Those of you who remember, know how the electric refrigerator almost put the ice manufacturer out of business; the important point is the ice-man did not fail—rather through the introduction of frozen foods the ice-man converted his equipment to flash freezing. Presto, he found himself right in the middle of a new, growing and profitable industry.

Frankly, I feel it is going to take a lot more than the loss of a few grease jobs to slow down our progress. Over the past ten years, the lubricating grease industry has made greater advances in all phases of the business than at any time during the past 100 years. The greaseless chassis bearing is just the prod we need to fight back with better lubricants, better marketing practices and better product realization. It also points up the need for finding new uses for grease mixes such as the manufacture of semi-solid rust preventives, caulking compounds, paints, plaster patch compounds and possibly plastic materials, etc. No doubt some of our more enterprising members are already investigating other products and newer markets. Like the ice-man of the late 20's, there is every reason to believe we will surmount this economic change and be better for it.

Ten thousand hours is a long study period... but it's a study period repeated year after year at **SOWESCO**... resulting in a sustained productive laboratory research program that makes available to our customers the most complete selection of customized, modernized lubricants ever available to branded marketers.

This 10,000-hour study period involves a never-ending search for saleable products... products tailored to solve your problems, be they problems of performance, packaging, or price.

Yes, a 10,000-hour productive, successful annual research program is another reason why we at **SOWESCO, the HOUSE of "good" GREASE** recommend that progressive branded marketers should Look Behind the Product for **"THE HIDDEN VALUE FACTORS."**

There's only ONE Leader

Your inquires will be enthusiastically received and handled.

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Future Meetings

JANUARY, 1961

9-13 Society of Automotive Engineers Annual Meeting, Cobo Hall and Convention Arena, Detroit.

FEBRUARY, 1961

5-10 ASTM Committee D-2 meeting, Benjamin Franklin Hotel, Philadelphia, Pa.

15 NLGI Board of Directors meeting, Sheraton-Cadillac Hotel, Detroit.

McGEAN 30% LEAD NAPHTHENATE ADDITIVE

- Consistently uniform in metallic content and viscosity
- Fully clarified by filtration
- Non-Oxidizing---contains no unsaturated soaps
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APRIL, 1961

11-13 American Society of Lubrication Engineers Annual Meeting and Exhibit, Bellevue-Stratford Hotel, Philadelphia.

19-20 National Petroleum Association, Semiannual Meeting, Sheraton-Cleveland Hotel, Cleveland.

MAY, 1961

8-9 ASME Lubrication Symposium, Deauville Hotel, Miami Beach, Fla.

13 NLGI Board of Directors meeting, Hollywood Beach Hotel, Hollywood Beach, Fla.

16-19 API Division of Marketing, Midyear Meeting, Americana Hotel, Miami Beach, Fla.

JUNE, 1961

11-15 ASME Summer Annual Meeting, Statler Hilton Hotel, Los Angeles.

24-29 ASTM Committee D-2 meeting, headquarters undecided, Atlantic City, N. J.

SEPTEMBER, 1961

11 NLGI Board of Directors meeting, Roosevelt Hotel, New York City.

13-15 National Petroleum Association, Annual Meeting, Traymore Hotel, Atlantic City.

OCTOBER, 1961

17-19 ASME-ASLE Lubrication Conference, Hotel Morrison, Chicago.

OCT. 29 - NOV. 1, 1961 NLGI Annual Meeting, Rice Hotel, Houston, Tex.

NOVEMBER, 1961

9-10 SAE National Fuels and Lubricants Meeting, headquarters undecided, Houston, Tex.

13-15 American Petroleum Institute Annual Meeting, headquarters undecided, Chicago.

26-Dec. 1 ASME Winter Annual Meeting, Statler Hilton Hotel, New York.

HARSHAW LEAD BASE

Harshaw Lead Base, as an additive to petroleum lubricants, improves extreme pressure characteristics and imparts the following desirable properties:

- Increased film strength
- Increased lubricity
- Improved wetting of metal surfaces
- A strong bond between lubricant and metal surfaces
- Resistance to welding of metals at high temperatures
- Moisture resistance and inhibits corrosion

Harshaw Lead Bases are offered in three concentrations to suit your particular needs:

Liquid 30% Pb	Liquid 33% Pb	Solid 36% Pb

Other metallic soaps made to your specifications. Our Technical Staffs are available to help you adapt these products to your specific needs.

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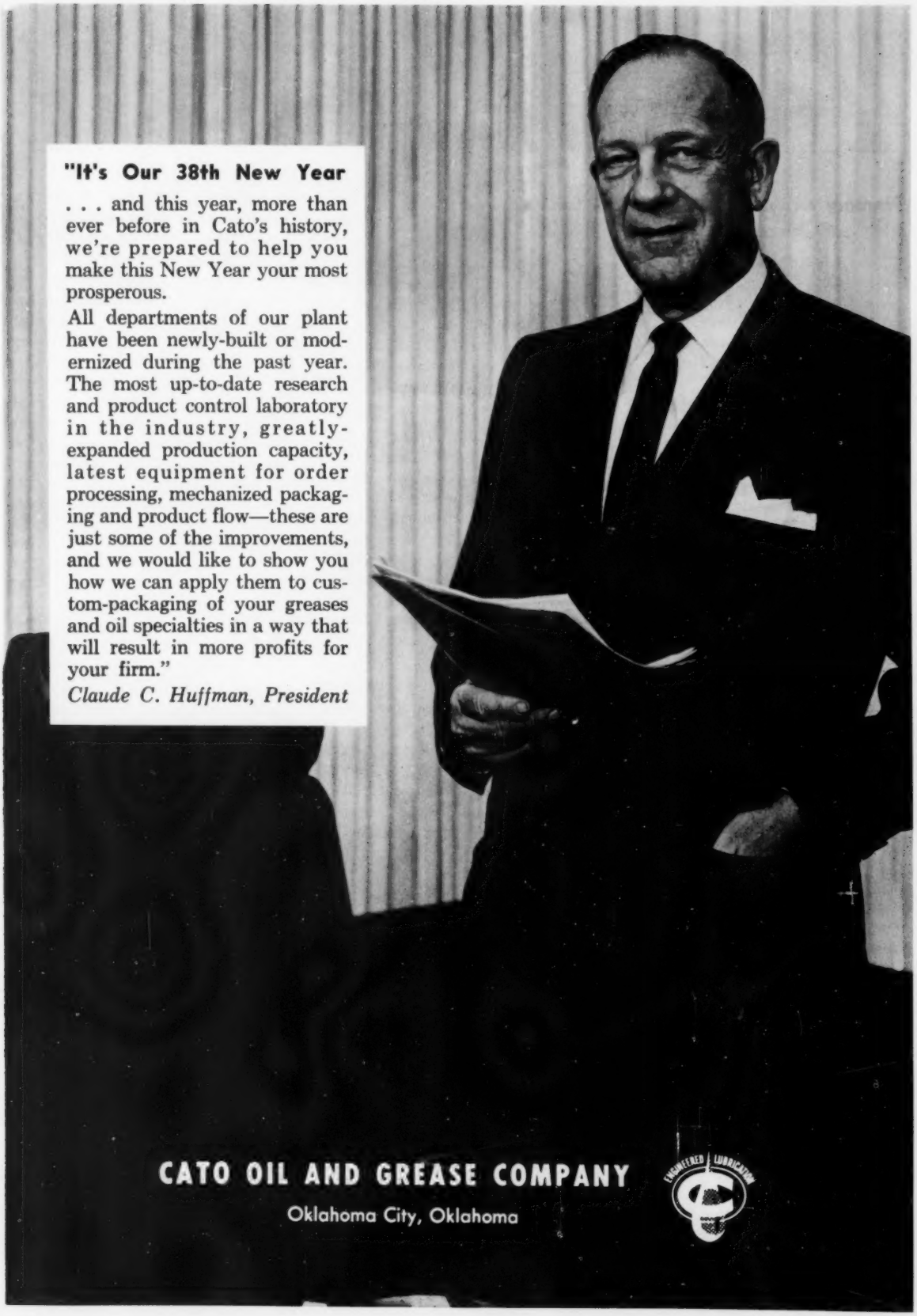

"It's Our 38th New Year
. . . and this year, more than ever before in Cato's history, we're prepared to help you make this New Year your most prosperous.
All departments of our plant have been newly-built or modernized during the past year. The most up-to-date research and product control laboratory in the industry, greatly-expanded production capacity, latest equipment for order processing, mechanized packaging and product flow—these are just some of the improvements, and we would like to show you how we can apply them to custom-packaging of your greases and oil specialties in a way that will result in more profits for your firm."
Claude C. Huffman, President
CATO OIL AND GREASE COMPANY
Oklahoma City, Oklahoma
ENGINEERED LUBRICATION

News About NLGI

Summary of Technical Committee Meeting Held in Chicago, November 2, 1960

The summary of the Technical Committee meeting held in Chicago, November 2, 1960, was prepared by Vice-Chairman M. L. Carter. Complete minutes will be distributed by the NLGI office.

Reports of Subcommittees

Steering Committee—L. C. Brunstrum, Chairman

Mr. Brunstrum reported that much had been done this year to facilitate communications. This has been done, not only within the Technical Committee, but also to the Board of Directors.

To improve the communications within the NLGI organization, the following has been planned:

1. A summary of the Technical Committee meeting is to be prepared and placed in the NLGI SPOKESMAN as quickly as possible.
2. A long form will be prepared later and sent to all members of the Technical Committee. Along with these minutes will be a request for comments and approval. It is urged that you offer your comments to complete the communications cycle.
3. Each committee chairman will furnish a midyear written report covering committee activities. This will be used for reporting to the Board and also for publishing in the NLGI SPOKESMAN.
4. Communications were also improved by assigning definite space and time for individual committee meetings during the conventions. Everyone agreed that this was an improvement.
5. Preprints of one technical session were distributed prior to the meeting to stimulate discussion of the papers. This worked out so well that discussions continued until all the time allowed had passed. This same policy will be tested again next year.

Larry then announced that Vice-Chairman Mike Pohorilla had resigned and that a new vice-chairman would be announced soon. In recognition of Mike's efforts the past several years, he was asked to conduct the rest of the program.

Gear Lubricants—R. K. Smith, Chairman

The past year was spent in developing papers on gear lubricants that could be given at the 1960 meeting. Due to gear lubrication symposia sponsored by other technical societies, this committee was unable to have papers presented as they expected. It is believed that a good symposium on gear lubricants can be prepared and presented at our 1962 or 1963 annual meeting. The need for closer liaison work between NLGI and other societies interested in problems of gear lubrication was again pointed out.

Consistency Projects—Melville Ehrlich, Chairman

The chairman pointed out the present NLGI classification was first published in the NLGI SPOKESMAN in January, 1940. Since then, there have been no changes even though this classification does not cover the fluid greases that most of the companies manufacture and market.

The second item pointed out by the chairman deals with the changes in the ASTM cone. Penetration may now be taken with either of two cones. The old one allows a penetration up to 400, while the new alternate cone, called the modified ASTM cone, may be used up to 475 penetration. When using the new cone, the old penetrometers may have to be altered to give satisfactory results. When using either cone, the tips should be carefully checked; as it has been found that among 26 tips checked, an eleven-point range was found. It is felt that if this range were cut down, much better checks between laboratories would result. The new specification calls for tip length of 0.588 ± 0.002 inches. It is recommended by the committee that each manufacturer or consumer making penetrations check tips and purchase new ones with the specification limits listed above.

The third item reported upon deals with grades for the fluid lubricants. It is believed that the range from 385 to 475 should be covered with two grades with customary gaps between them. We should also give some thought to the grade designations such as 00, 000; or 2-0, 3-0; or F2, F3, etc. Comments on this subject will be appreciated.

Recommended Practices—P. V. Toffoli, Jr., Chairman

This subcommittee believes that a survey should be made of the various grease manufacturers and solicit data showing correlation to lubrication interval and wear. With proper data, the automotive companies could be approached on this subject.

A second project under consideration is the revision of the wheel bearing procedures. Along

Continued on page 392

Better Greases Through BAROID RESEARCH

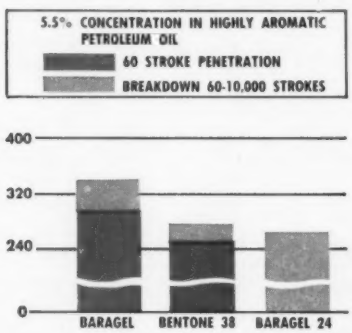
BARAGEL* 24 stabilizes greases made from petroleum oils

LABORATORY REPORT

BARAGEL* 24 in grease made from high aromatic oil

In a particular petroleum oil of high aromatic content (28%), BARAGEL 24 produced a grease completely stable to working in the ASTM grease worker over the range of unworked to 10,000 strokes.

In a series of tests, the oil used had an API gravity of 16.9° at 60° F and a viscosity of 309 SUS at 100° F. The worked penetration was 265 when

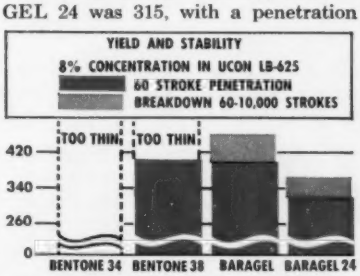


5.5% BARAGEL 24 was used. Comparisons of penetration when using the same percent of BENTONE* 34 and BARAGEL* are shown on the accompanying chart. Write to Baroid Chemicals, Inc. for Data Sheet A-4 which gives full details.

BARAGEL* 24 efficient thickener in greases made with Ucon LB-625

BARAGEL 24 is more efficient in thickening Ucon LB-625 than are BENTONE 34, BENTONE* 38 and BARAGEL. BARAGEL 24 greases are also more stable to working in the ASTM grease worker.

In a series of tests, the gelling agents were stirred into the Ucon LB-625, propylene carbonate was added, heat applied, and the mixture was milled. The chart below compares the results of thickening greases with BENTONE 34, BENTONE 38, BARAGEL and BARAGEL 24. The worked penetration of a grease made from Ucon LB-625 and 8% BARAGEL 24 was 315, with a penetration



of 355 after working 10,000 strokes. Full test details are available in Data Sheet A-1. Write Baroid Chemicals, Inc.

*Trademark of National Lead Company for an organic ammonium montmorillonite.

BAROID

BAROID CHEMICALS, INC.

A SUBSIDIARY OF NATIONAL LEAD COMPANY 1505 SOUTH COAST BLDG. · HOUSTON 2, TEXAS

News About NLGI cont'd

these same lines, a suggested procedure for lubricating these new so-called sealed bearings (used for steering and suspensions where plugs are used) might be in order. Any suggestions covering this subject will be welcome.

Central Dispensing Systems—C. F. Raisch, Chairman

This is a new committee organized the past year and is composed of one representative from five grease manufacturers, four from equipment suppliers and one user. This committee is to study the dispensing problems in centralized systems. It has been found that the shear rates in these systems are much lower than those shear rates previously worked with. Some type of test equipment is to be set up to determine if reproducible results can be obtained. Later, these results will be correlated with the apparent viscosity of the grease sample.

Manual of Test Methods and Definitions—Albert S. Orr, Chairman

There is a strong feeling that the manual on test methods should be dropped, mainly because it is essentially paraphrasing ASTM test methods. Unless there is some serious objection, no further work will be done on test method write-ups. A new list of definitions has been made up, and it was proposed to circulate these for approval by letter ballot about December 1, 1960.

Editorial Review—George Entwistle, Chairman

The chairman summarized the activities of this subcommittee as follows:

1. Twenty-six papers were reviewed.
2. Twenty papers were approved.
3. Three papers were turned down.
4. Suggested changes were made to authors of three other papers.
5. An attempt is being made to present a session at the next annual meeting, dealing with editorial matters.

Procurement of Papers for Publication in the NLGI *Spokesman*—C. J. Boner, Chairman

It was pointed out that the annual meeting supplies about two-thirds of the articles to fill the space in the NLGI SPOKESMAN. This indicates a very minimum of 12 additional papers needed for 1961. It is urged that you keep the journal in mind as a means of publicizing the results of your technical results. An additional project has been the listing of symbols to be used in writing and printing technical articles in the magazine.

Manufacturing Operations—J. J. Dickason, Chairman

Papers were solicited in behalf of NLGI for the joint ASTM-NLGI symposium on the subject, "The Significance of Oil Viscosity in Lubricating Grease," to be presented in Houston next year. Four papers have already been promised and two more are requested. One of these is preferred to be from a user of lubricating greases, showing the need of oil viscosity ranges in grease specifications.

Fundamental Research—E. L. Armstrong, Chairman

This committee, which is one of the newer ones on the Technical Committee list, sponsored its second research symposium on structure and flow properties of greases on Tuesday morning. The first was held at the 1958 annual convention. This committee recommends the following for 1961:

1. The NLGI Fellowship at the University of Utah be continued for another year. This involves the testing, examination and evaluation of three grease systems now under study. These series include silica, bentone and calcium soap greases.
2. With respect to the second NLGI Fellowship, Dr. Vold's proposal for "A Fundamental Investigation Concerning the Interaction Among Thickener, Additive and Oil in Greases" be accepted.

It is also suggested that we continue our search and look particularly for people to study friction and wear characteristics of greases. It is thought that contact with the ASME Research Committee would be helpful—contact could be made with university professors in mechanical engineering schools who may be interested in and equipped to work on such problems.

It is also suggested that research papers and attendance at our meetings could be stimulated by enlarging the mailing list to interested people and helping other people obtain special information on grease research equipment.

Motions were made and passed that the present fellowship be continued and that the second fellowship offered by Dr. Vold be accepted.

Grease Incompatibility Study Group—M. J. Pohorillo, Chairman

In 1959 this group was assigned the task of spearheading an educational program to acquaint consumers with the problem of grease incompatibility. During the past year, work was started on the preparation of a descriptive publication which could be used by the NLGI in the form of a bulletin similar to those describing proper procedure for lubricating wheel bearings and ball joints. This study group earnestly solicits participation by all Technical Committee members in making available any sales training literature and laboratory or field test data that could be used in this program.

Your comments or suggestions covering any phase of the Technical Committee program are earnestly solicited.

now available *now available*

NLGI SPOKESMAN

VOLUME XXIII

Latest Issue of Institute's Journal In Bound Volumes Offered to Readers

A reference work which is both handsome and practical . . . Volume XXIII of the NLGI SPOKESMAN contains the twelve issues between April, 1959 and March of 1960 . . . 36 features on every phase of the lubricating and fluid gear lubricants industries. Some categories:

> Automobiles . . . automatic transmission fluids . . . fatty acids . . . manufacture . . . non-soap . . . non-soap thickeners . . . testing . . . gear lubricants . . . marketing . . . non-lubricated bearings . . . and others.

The sturdy green cover holds all twelve issues securely, with an index in the final magazine broken down by author, title and category. A few copies of Volume XXI and XXII are also available. Write NLGI, 4638 J. C. Nichols Parkway, Kansas City 12, Missouri. $7.50 for members, $10.50 for friends of NLGI, plus postage.

Volumes I through XXIII may be purchased from University Microfilms, 313 North First Street, Ann Arbor, Michigan.

NLGI Presents Its Governing Body for 1960-61

AN ASSOCIATION, a society, or an institute is a cooperative organization—usually representation is made up of a group of men in a particular industry—competitors in business, but working together voluntarily to promote their industry and business generally. Through a governing board, the membership reflects its aims and desires concerning policy and planning. But voluntary assistance is needed to enact the organization's work and in this respect NLGI is fortunate in that it has a "working" board—every member of the governing body is responsible for one or more facets of the operation, giving freely of time and effort to strengthen the Institute's objectives . . . "To develop better lubricating greases for the consumer, better grease lubrication engineering service to industry." Shown here are the officers and directors of the National Lubricating Grease Institute and their committee assignments for 1960-61.

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Publicity
Production Survey

T. W. BINFORD
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ASLE Liaison

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Chmn: Production Survey

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Chmn: API-NLGI Joint Container Committee

Subcommittee Chairmen of NLGI Technical Committee

For a summary of the latest Technical Committee meeting, see page 390.

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Sinclair Refining Co.
Editorial Review

M. J. POHORILLA
Kendall Refining Co.
Grease Incompatibility
Study Group

Presented at the NLGI 28th annual meeting in Chicago, October, 1960

Oxidation of Lubricating Greases

By: J. E. Goodrich and J. J. Burke
California Research Corporation

Introduction

An important property of lubricating greases is their level of oxidation resistance. For this reason, many grease specifications include a maximum allowable oxygen pressure drop in bomb oxidation tests. In spite of the importance of oxidation stability of greases, relatively little has been published on the subject. This is in contrast to the voluminous literature on lubricating oil oxidation.

A number of aspects of grease oxidation have attracted our interest. These include the factors which determine the shapes of oxidation curves and the effects of grease composition, such as soap and base oil type, and acidity or basicity. We studied the nature of grease oxidation products and developed spectroscopic techniques for detecting their presence. The results of this work shed new light on the little understood mechanism of grease oxidation and indicate the relative importance of some of the factors which influence it.

Our work was limited to a study of uninhibited mineral oil or hydrocarbon oil based greases, thickened with stearate soaps of alkali metals, alkaline earth metals, or aluminum. The bomb oxidations were all carried out at 210°F. The base oils included the following:

a. Cetane—commercial, untreated.
b. Neutral Oil—a solvent-refined 480 Neutral Oil from paraffinic base crude.
c. Pale Oil—a solvent-refined 450 Pale Oil from a naphthenic base crude.
d. White Oil—an oil meeting USP specifications for heavy liquid petrolatum, uninhibited.

Bomb Oxidation Test

The ASTM Bomb Oxidation Test (ASTM Method D 942-50) is the accepted method of measuring the oxidation stability of lubricating greases. In this test, twenty grams of grease is placed in glass dishes supported by a rack and inserted in a Norma-Hoffman oxidation bomb. The bomb is charged with oxygen, placed in a 210°F oil bath, and the change in oxygen pressure as a function of time is noted.

According to the ASTM procedure, the bomb is charged with 100 psi of oxygen at room temperature, tested overnight for leaks, and the pressure is then adjusted to 110 psi after the bomb is placed in the oil bath. Our procedure differs in that the bombs are charged with 90 psi of oxygen at room temperature, tested for leaks, and no adjustment of pressure is made after the bomb is placed in the bath. Using this method, the initial oxygen pressure at 210°F is near but not always equal to 110 psi. This method has the advantage that no adjustment of valves is necessary after the test for leaks has been completed. A photograph of our bomb rack fitted with pressure recorders is shown in Figure 1.

A typical plot of oxygen uptake is shown in Figure 2. Many greases and oils first exhibit an induction period (labeled A) during which oxygen uptake is slow. This is then followed by a period of rapid oxygen consumption (B) which is terminated by a period of essentially constant pressure (C). The magnitude of the induction period is arbitrarily determined by the intersection of the tangent to B with the tangent to A (shown by Point D in Figure 2).

Many materials oxidize at rates described by S-shaped curves, similar to Figure 2. However, the flat period (C) during which oxidation has apparently dramatically ceased is peculiar. At first it was postulated that at the end of Period B, the oxygen in the bomb had all been replaced by inert gaseous oxidation products, such as carbon dioxide, causing a halt in oxidation. To test this theory, a bomb was recharged with

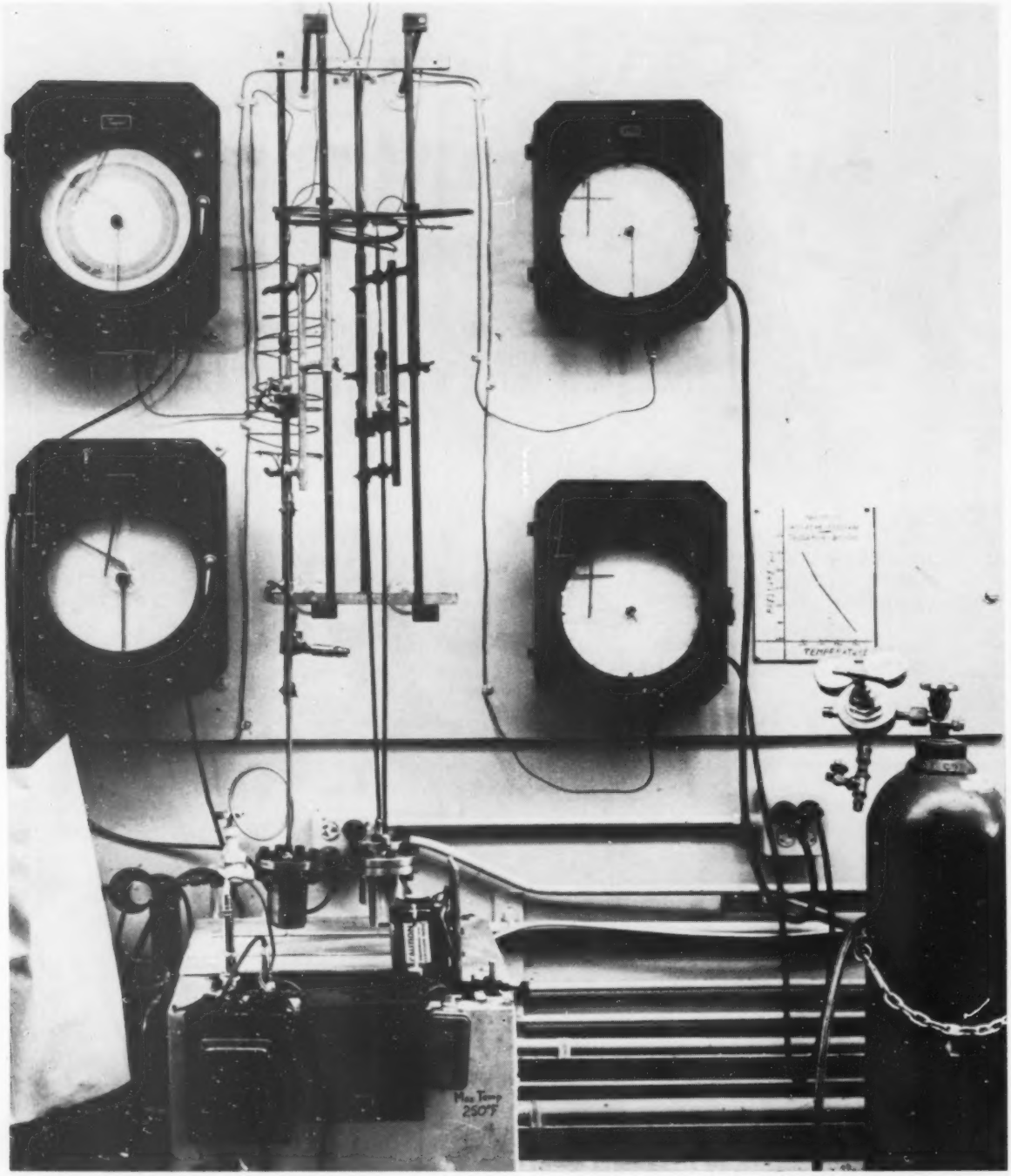

FIGURE 1—Bomb oxidation equipment.

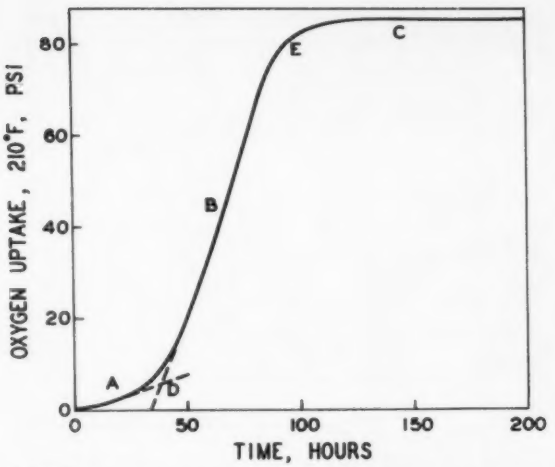

FIGURE 2—Typical grease oxidation plot. (15 weight per cent lithium stearate in neutral oil)

fresh oxygen after reaching Point E in the oxidation curve, and it was found that the grease continued to oxidize at about the same rate as Period B. When this procedure was repeated, recharging the bomb with fresh oxygen after reaching Point E a second time, the oxidation rate began to decrease. Figure 3 shows that when a composite plot is made of the oxygen uptake for the three runs, the grease exhibits a typical S-shaped oxidation curve.

To substantiate further our preliminary theory of oxygen depletion, the residual gases in the bomb present after 137 hours of oxidation (flat portion of curve in Figure 2) were analyzed by means of the mass spectrograph. Surprisingly enough, about one-third of the gas was oxygen. However, when the residual gas in a repeat test was analyzed after 283 hours of oxidation, the oxygen content had dropped to 14 per cent. The bulk of the remaining gas was carbon dioxide. These data, shown in Table I, indicate that oxidation has not stopped at Point E, but that the rate of gas formation (mostly carbon dioxide) just about equals the rate of oxygen consumption; and the total pressure remains essentially constant. Therefore, during Period C, oxidation is still continuing, but at a relatively slow rate because of the decreasing partial pressure of oxygen.

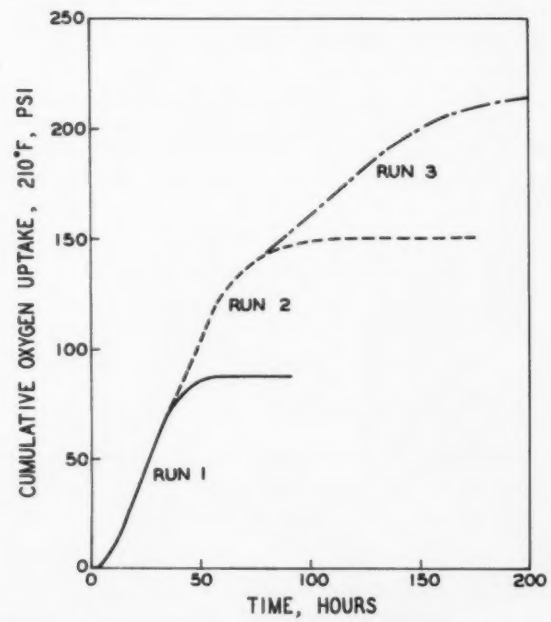

FIGURE 3—Successive oxidations of same grease. (15 weight per cent lithium stearate in white oil)

TABLE I

Composition of Residual Gas

(Oxidation of 15 weight per cent lithium stearate in neutral oil, 210°F.)

	Volume Per Cent	
Duration of Oxidation	137 *Hours*	283 *Hours*
Component:		
Oxygen	33.6	14.0
Carbon Dioxide	56.5	72.9
Carbon Monoxide	3.7	5.9
Hydrogen	1.3	1.7
Water *	1.0	0.6
Other (by difference)	3.9	4.9
	100.0	100.0
Total Pressure, psi	24	22

* *Vapor phase only. Additional water is found in the oxidized grease.*

Effect of Grease Composition

Soap

It is well known that uninhibited greases have poorer oxidation stability than the constituent mineral oils. This suggests that the soap gelling agents catalyze the oxidation of the oil. Bondi, et al.[1] found that dissolved soaps have no catalytic effect, and they therefore concluded that with insoluble soaps the catalysis takes place at the soap-oil interface. They suggested that the soaps accelerate oxidation of oils by selectively absorbing the natural inhibitors contained in the oils.

Two experiments were carried out in our laboratory to demonstrate soap catalysis. In one, a lithium stearate aerogel was used as the soap. This material, prepared according to the techniques described by Peterson and Bondi,[2] has the same gel volume as the soap in a grease, but is oil free. At first an attempt was made to oxidize the aerogel and mineral oil separately. Neither reacted with oxygen alone (less than 2 psi oxygen drop in both cases during 200 hours); but when the aerogel was added to the oil (at 15 weight per cent concentration) and oxidized, the mixture was highly unstable, as shown

in Figure 4. This mixture oxidized at essentially the same rate as a mineral oil grease thickened with 15 weight per cent lithium stearate formed *in situ* (compare Figure 2). This showed that the two grease constituents, soap and oil, were stable alone but unstable when mixed.

In the other experiment, a lithium stearate-neutral oil grease was first oxidized to determine its oxidation rate. Then, a fresh sample of the same grease was oxidized, but the test was halted at the end of the induction period. The grease was removed from the bomb and filtered. The mineral oil filtrate, free of soap, was then recharged to the bomb; the bomb was repressured with oxygen, and the test was continued. However, the oil was now stable and took up no more oxygen during the remaining 135 hours of the test. The

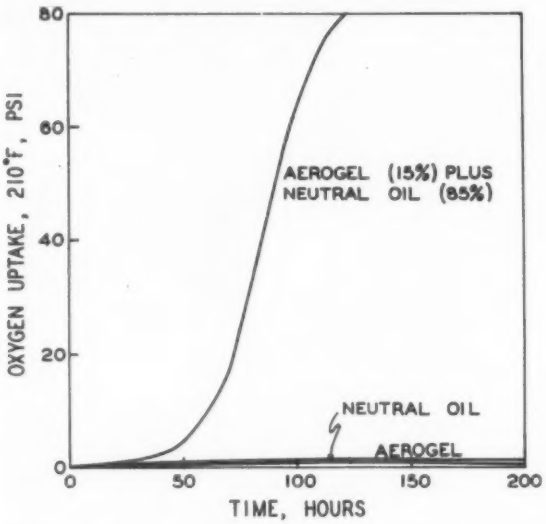

FIGURE 4—Oxidations of lithium stearate aerogel and neutral oil.

oxidation curves are shown in Figure 5. This experiment demonstrated that the soap catalyzes the oxidation of the mineral oil, but that if the soap is removed during the course of oxidation, the mineral oil regains its original high resistance to oxidation.

No investigations of the relative catalytic activity of the various alkali and alkaline earth metal soaps have previously been reported. Therefore, greases from various stearate soaps were prepared in neutral oil, and their oxidation stabilities were determined. All of the greases were based on 15 weight per cent stearic acid, and stoichiometric amounts of base were used to form the soaps. In addition, an aluminum stearate grease was prepared in neutral oil from aluminum isopropoxide.

These greases exhibited large differences in oxidation rates, depending upon the composition of the soap. The

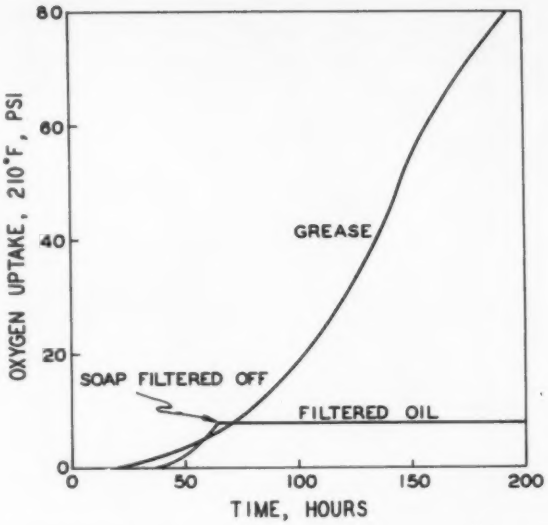

FIGURE 5—Effect of soap removal on grease oxidation stability. (15 weight per cent lithium stearate in neutral oil)

soaps ranged in activity from lithium stearate, the most powerful catalyst, to aluminum stearate, the mildest. The results are shown in Table II, in which the induction periods in hours are tabulated according to the position in the periodic chart of the metal in the soap. The numerical values of these induction periods are not absolute, but are subject to repeatability variations of about ± 10 per cent. However, the general decrease in catalytic activity going down in the periodic table, and to the right, suggests a chemical mechanism of soap catalysis.

To determine the effect of soap concentration on catalytic activity, a series of calcium stearate-neutral oil greases was prepared, ranging in soap content from

TABLE II

Catalytic Effect of Soaps

(15 weight per cent stearate soap in neutral oil)

Induction Period at 210°F, Hours		
Group I	*Group II*	*Group III*
Lithium 25		
Sodium 45	Magnesium 112	Aluminum 330
Potassium 158	Calcium 100	
	Barium 135	

3 per cent to 15 per cent. As shown in Figure 6, the catalytic activity increased as the soap concentration increased. The induction period of 1600 hours for the mineral oil alone was reduced to less than 300 hours with 3 per cent soap, and to less than 100 hours with 15 per cent soap.

Base Oil

The degree of refinement and chemical nature of the base oils affect the oxidation stability of greases. Macdonald and Dreher[3] found that the higher the degree of refinement of mineral oils (from the same crude source), the more susceptible were their greases to oxidation. Light treatment, with acid or solvent, removes the more readily oxidized materials and improves oxidation stability; but heavier treatment removes natural inhibitors and decreases stability.

To compare the effects of base oil type, lithium

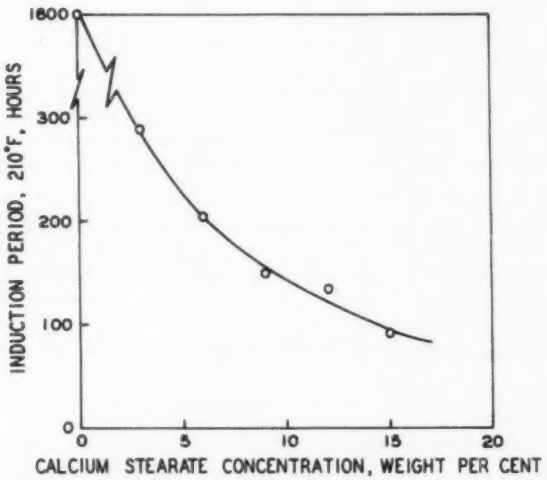

FIGURE 6—Effect of soap concentration—calcium stearate in neutral oil.

stearate greases made with neutral oil, white oil and cetane were oxidized. The oxidation rates of the base oils alone were also measured. Figure 7 shows that the oils increased in stability going from white oil to cetane to neutral oil, and likewise the stabilities of their lithium stearate greases increased in the same order. The greases were all less stable than their base oils. The fact that lithium stearate catalyzes the oxidation of cetane, which of course contains no natural inhibitors, disfavors the Bondi postulate that soaps catalyze oxidation by absorbing natural inhibitors from the oil.

Acidity and Basicity

Alkali and alkaline earth hydroxides have long been known to retard the oxidation of mineral oils and greases. During oxidation, acidic materials are formed; and presumably the bases neutralize these acids and

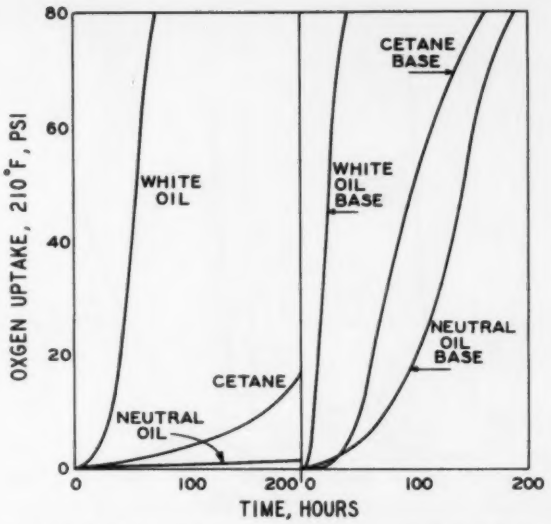

FIGURE 7—Effect of base oil on grease oxidation stability. L., base oils alone; r., 15 weight per cent lithium stearate greases.

slow down the oxidation rate.

A quantitative measure of the effect of excess alkalinity and acidity was found by studying three lithium stearate-neutral oil greases, all based on 15 weight per cent stearic acid. The amount of lithium hydroxide used was 75 per cent, 100 per cent, and 125 per cent of the theoretical amount necessary to neutralize the stearic acid. Oxidation curves of the three greases are

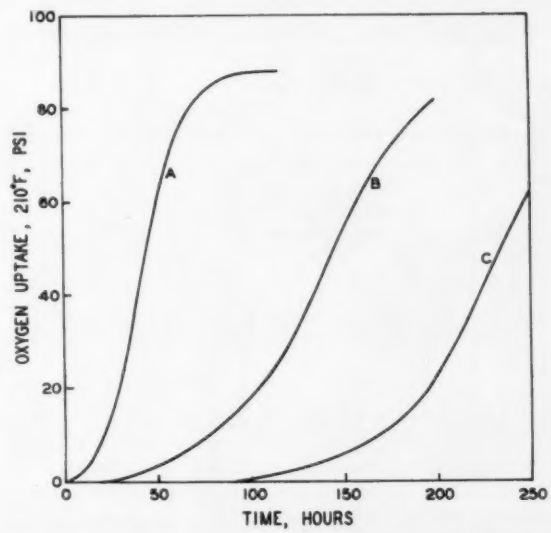

FIGURE 8—Effect of grease acidity and basicity. Lithium stearate in neutral oil (based on 15 weight per cent stearic acid). Per cent of theoretical lithium hydroxide used: A—75%; B—100%; C—125%.

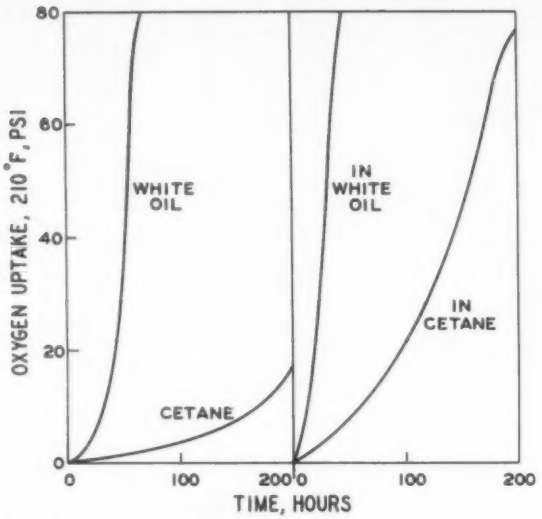

FIGURE 9—Effect of stearic acid on oxidation stability. L., base oils alone; r., 15 weight per cent stearic acid.

shown in Figure 8, demonstrating that excess base retards oxidation; excess acidity accelerates it.

In another series of experiments, it was found that stearic acid alone (at 15 weight per cent) accelerates the oxidation of white oil and cetane. This is shown in Figure 9. No catalytic effect of stearic acid in neutral oil was observed; however, the test was discontinued after 208 hours, and the induction period of the mixture may well have been less than the 1600-hour induction period of the mineral oil alone.

Identification of Oxidation Products

In oxygen bomb tests, greases react with oxygen to form gaseous and nongaseous products. The composition of the gases was found with the mass spectrograph and the others with the infrared spectrophotometer. During oxidation, greases form a band in the 1710-1740 cm^{-1} range of the infrared spectrum. This is the region of carbonyl absorption, characteristic of aldehydes, ketones, esters and acids. Soap carbonyls do not interfere, as they absorb at 1550-1610 cm^{-1}, outside of the spectral region of the oxidation products.

A useful technique in studying the infrared spectra of greases during oxidation is to spread thin films of greases on the salt windows used in the infrared spectrophotometer. These windows can be placed in the racks in oxygen bombs, and the grease films can be oxidized in the same manner as grease in glass dishes. Spectra of greases may be obtained both before and after oxidation by inserting the salt windows directly in the spectrophotometer.

Comparison of infrared spectra of a grease before and after oxidation is shown in Figure 10. It shows the large increase in concentration of carbonyl compounds in the 1710 cm^{-1} region during oxidation. The increase in intensity of the 3500 cm^{-1} band indicates the formation of hydroxyl groups, probably due to water and carboxylic acids.

To find whether the oil or the soap was the source of oxidation products, the soap was filtered from an oxidized grease and examined in the infrared spectrophotometer. The soap had essentially no absorption in the 1710-1740 cm^{-1} region, but the filtered oil absorbed strongly in this region. This indicates that the

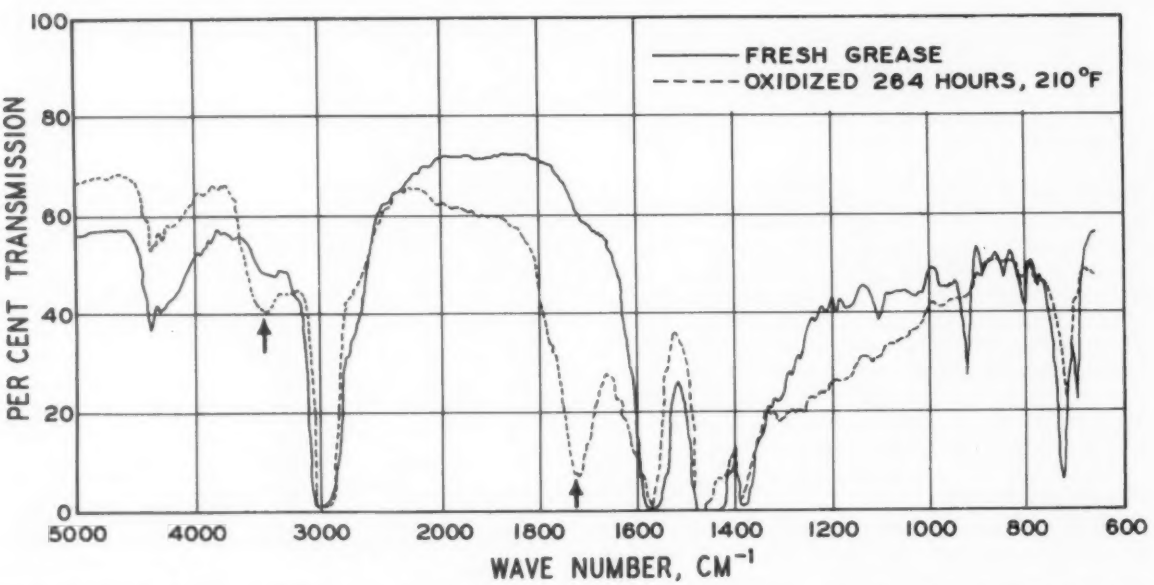

FIGURE 10—Infrared spectra of grease before and after oxidation. (15 weight per cent sodium stearate in neutral oil)

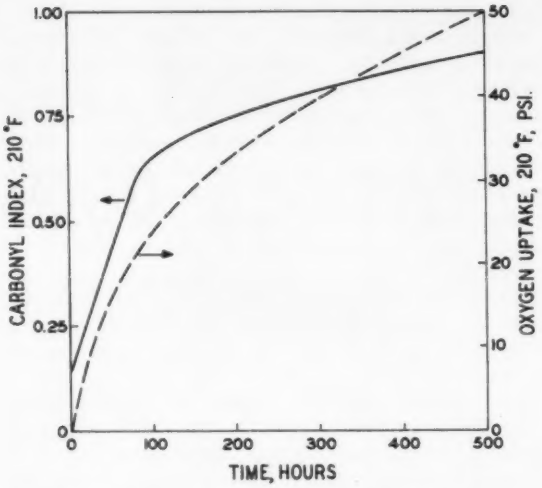

FIGURE 11—Comparison of Carbonyl Index and pressure drop during grease oxidation. (12 weight per cent lithium stearate in pale oil)

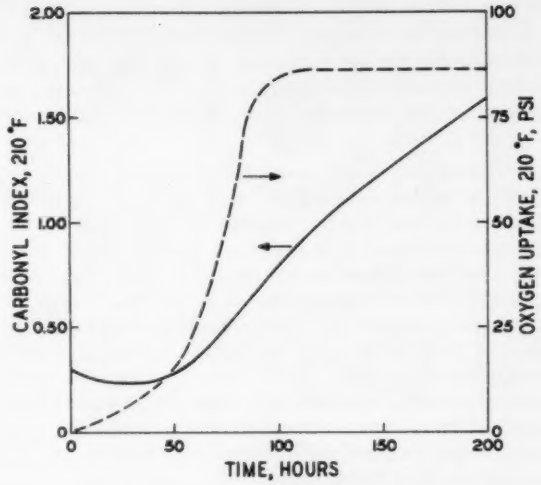

FIGURE 12—Comparison of Carbonyl Index and pressure drop during grease oxidation. (15 weight per cent lithium stearate in neutral oil)

oxidation products are in the oil phase, but does not rule out the possibility of the formation of oil-soluble oxidation products from oxidative attack on the soap.

The infrared spectrophotometer can also be used to determine the degree of oxidation of a grease. Quantitatively, this is measured in terms of the Carbonyl Index, defined as the ratio of the absorbance of the carbonyl band at 1710 cm^{-1} and the absorbance of the methyl band at 1378 cm^{-1}. Use of the methyl band absorbance as a reference compensates for variations in grease film thickness.

Figures 11 and 12 are plots of the change in Carbonyl Index and bomb pressure drop during the oxidation of two greases. Figure 11 shows marked similarity in the shapes of the two curves for a pale oil grease. The curves for the neutral oil grease in Figure 12 differ in that bomb pressure drop halts after 100 hours of oxidation, yet the Carbonyl Index curve continues to rise. This confirms the results of the gas analysis study described earlier, indicating that oxidation continues to occur even after pressure drop ceases.

By noting the change in Carbonyl Index as a function of time, the rate of oxidation of a grease can thus be found. The Carbonyl Index is also a convenient measure of degree of grease oxidation during manufacture, during service, or in storage.

Evidence of the chemical nature of the carbonyl compounds which form during bomb oxidations was obtained by chemical and spectrographic analysis of oxidized white oil. The oxidized material consisted of an oil layer and an acidic aqueous layer. The organic acids were first extracted with sodium bicarbonate solution from the oil phase. This reduced the intensity of the carbonyl band somewhat, but still left the bulk of the carbonyl compounds in the oil. The oil was then warmed with dilute sodium hydroxide to hydrolyze any esters present. This treatment almost eliminated the carbonyl band from the oil phase, indicating that esters are the predominant oxidation products, but that small amounts of aldehydes or ketones may be present. The acidic fractions originally present in the oil and aqueous phases, and those liberated by ester hydrolysis were chiefly formic, acetic and propionic acids, according to the infrared spectra. The alcohols which combined with these acids to form esters were found spectroscopically to have carbon chains greater than C_4.

Previously, it was found that carbon dioxide and carbon monoxide are the chief gaseous products of grease oxidation. The above analyses show that acids, esters and water are the other chief oxidation products of white oil. Some low molecular weight aldehydes or ketones may have been present in the aqueous phase, but only negligible amounts of higher molecular weight aldehydes or ketones were found in the oil phase. We have not analyzed any oxidized greases for nongaseous products, but predict that they are similar in composition to those in oxidized white oil.

Discussion of Results

It would be desirable to relate the results of bomb oxidation tests to the service performance of greases. No such relationship has yet been established. The scope of the bomb oxidation test, as stated in the ASTM Manual is as follows: "This method describes the test for determining the resistance of lubricating greases to oxidation when stored under static conditions for long periods of time as, for example, thin coatings on antifriction bearings and on motor parts, etc.

It is not intended for the prediction of the stability of greases under dynamic service conditions, nor is it intended for the prediction of the stability of greases stored in commercial containers." This indicates that at present the functionality of the bomb oxidation test is quite limited.

With regard to the mechanism of grease oxidation, there is abundant evidence that soaps of metals of Groups 1, 2 and 3 in the periodic chart catalyze hydrocarbon oxidation. This is evident from our own work and also from information from other fields. Potassium, calcium, barium, magnesium and aluminum soaps are used commercially as oxidation catalysts in the production of fatty acids by hydrocarbon oxidation. Barium naphthenate has been used as a catalyst for the oxidation of paraffin wax. Calcium soaps are common oxidation catalysts in paint dryers. Sodium, potassium, magnesium and calcium naphthenates all catalyze the peroxidation of hydrocarbons.

Yet none of the above soaps, made from metals with only one valence state, fit the classical concept of oxidation catalysts. Salts or soaps of transition metals (such as copper, cobalt, manganese, iron and lead) are typical oxidation catalysts. The efficiency of these catalysts is thought to depend upon the ability of their metallic constituents to change valence.

There is yet no satisfactory explanation why soaps of alkali and alkaline earth metals with only one valence state are oxidation catalysts. It is true that their catalytic activity is of a different order of magnitude from that of the variable valence metal soaps. Several per cent of alkali or alkaline earth metal soap are equivalent in activity to several parts per million of transition metal soap. This indicates that different mechanisms may operate for the two types of soaps.

One suggested explanation of the catalytic effect of soaps of metals with only one valence state is that metals with variable valence are present in these soaps as impurities.[5] Our work showing the relation between catalytic activity and location of the metal in the periodic chart indicates that there is a systematic chemical basis for the catalysis which is not due to trace impurities.

An investigation of the mechanism of soap catalysis should prove a fruitful field of research. The results would be of value not only in the lubricating grease field but also in the many other fields where soaps play roles as oxidation catalysts.

It should be mentioned that although soaps catalyze hydrocarbon oxidation, the catalytic activity can be readily counteracted by a variety of oxidation inhibitors. These inhibitors, including aromatic amines and phenols, dialkyl selenides and some heterocyclic compounds, are widely used in lubricating greases and provide oxidation protection for marketed products.

Acknowledgment

The authors wish to thank Mr. H. M. White for developing the techniques for examining greases in the infrared spectrophotometer, and for originating the Carbonyl Index method for determining the degree of grease oxidation.

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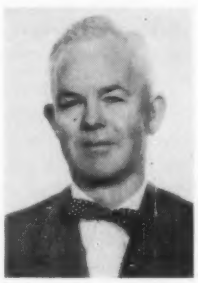


THE 1958 FOOD ADDITIVES AMENDMENT

Its Effect on The Lubrication of Food Packaging Machinery

By: A. E. Dooley
Texaco Inc.

Presented at the NLGI 28th annual meeting in Chicago, October, 1960

A YEAR AGO, even six months ago, it would have been more difficult to discuss this subject. But now things are much clearer. As John L. Harvey, Deputy FDA Commissioner, put it in a talk to the Technical Association of the Pulp and Paper Industry last June, "The seas grow calmer."

When the Food Additives Amendment was passed two years ago, food manufacturers for the most part were generally aware of its provisions. Manufacturers of food packaging materials were less informed of the possible application of the law until about May or June, 1959. Then a real flood of inquiries started. For a while it seemed that every manufacturer of a product used in or around a food plant or a packaging material plant was getting letters from his customers asking if his products had been cleared by the FDA and, if not, what steps were being taken to obtain clearance. Some of the inquiries at the time seemed far-fetched. One that comes to mind was whether kerosene had been approved as a food additive by the FDA. It was from a paper manufacturer who used kerosene to de-ink waste newspapers prior to pulping them to make paper. He was, in all good faith, concerned because some of his paper might be used to wrap food. While most of the inquiries dealt with non-lubricating uses, enough had to do with lubricants to indicate that there was considerable confusion as to whether oils and greases were food additives.

To show the position of the lubricant manufacturer in the food additive picture, it would be well to tell what a food additive is. It will be easier to define a food additive in a negative way. Thus the following are *not* food additives:

a. Substances generally recognized as safe.
b. Certain pesticides used on raw agricultural products.
c. Substances used in foods under approval or sanction prior to the passage of the Amend-

ment, or under the Poultry Products Inspection Act or the Meat Inspection Act.

d. Substances, the intended use of which may not reasonably be expected to become components of the food or otherwise affect the characteristics of the food.

The law does not define "reasonable". Mr. E. T. Wulfsberg has stated that the FDA interprets it as "equitable, fair and considerate of both sides of a question". All other substances become food additives under the law when they get into food, directly or indirectly. The Amendment does not cover accidental contamination of a food. The FDA had the authority to act in such cases long before the 1958 Amendment.

From this definition it can be seen that the place of the lubricant manufacturer in the food additive picture is, at best, a minor one. He has no direct responsibility for the safety of a food. He makes and sells his lubricating oils and greases for the lubrication of machinery and he does not intend them to become components of food. Furthermore, he normally has no control over how his products are used.

A review of the regulations and the petitions published in the Federal Register shows that consideration of petroleum products at least up to now relates to their non-lubrication uses.

No oils or greases are listed among substances generally accepted as safe or among the materials which the Commissioner proposes to list as safe. The FDA points out that the absence of a material from this so-called "white list" does not necessarily mean that a material is unsafe. Nor is any lubricating oil or grease named among the materials for which sanction for use had been granted prior to enactment of the Food Additives Amendment. Furthermore, of the approximately 25 approvals granted so far, none deals with a petroleum product. Only one material has been approved as an equipment lubricant. It is distilled acetylated monoglycerides and not over 5 per cent is permitted in food. This material obviously is not a conventional lubricant.

Only two formal petitions for petroleum products have been published to date and both have been withdrawn without prejudice. One of these requested a residue tolerance for kerosene or mineral oil on metal foil when these materials are used as lubricants in foil manufacture. The other dealt with mineral oil as a constituent of a food wrap. Other petitions have been filed but in effect they were requests for extensions and have been included in the lists of extensions for use until March 6, 1961.

Extensions have been given for the following petroleum products: (The terms are those listed in the Federal Register.)

Wax, microcrystalline and paraffin—as a coating for fruits, vegetables and cheese; as a masticating agent in chewing gum, as a constituent of food packaging materials, as a lubricant or release agent; as a migrant from container linings when used as a release agent or surface lubricant; as a migrant from cotton and cotton fabrics.

Mineral oil (including petrolatum)—as a lubricant or dust control agent in bulk and pelleted animal feed including salt blocks; as a protective coating on fresh fruits or vegetables; as a lubricant in meat packing plants; as pan oil, dough divider oil or trough grease in bakeries; as a release agent in drying pans for egg albumin, dried fruits and vegetables; as a migrant from cotton and cotton fabrics; as a release agent or surface lubricant ingredient in container linings; as a migrant from packaging material; as a secondary plasticizing agent in rubber belting, hose or rollers having food contact; in fiber bags to contain dry food; as a constituent of defoaming agents in the manufacture of beet sugar.

Aliphatic kerosene—as a component of defoaming agents in the manufacture of beet sugar.

Naphtha—as a migrant from adhesives.

Defoaming agents migrating from paper and paperboard products—aliphatic hydrocarbon fractions of petroleum, completely aliphatic kerosene, kerosene oil, deodorized kerosene oil, mineral oil, naphtha, paraffin oil, pale paraffin oil, paraffin wax.

Specifications have been published only for microcrystalline and paraffin wax. Specifications for the mineral oil and petrolatum were not published. However, it is generally known that the mineral oil and petrolatum extensions apply only to the USP grades of these products plus an ultraviolet absorbance requirement. These grades authorized under the extension are not generally considered as lubricants.

Lubricant manufacturers have no immediate part in the manufacture, processing or packaging of a food. But their customers do. They may be food processors, food packagers or manufacturers of packing materials. Between the two groups are the designers and builders of the machinery. Each group has his own responsibility in the food additive picture.

The food processor or packager has the direct responsibility for the safety of his product. He, therefore, must operate his plant so as to prevent the entry into food of any unauthorized component. If the machinery he uses is designed and maintained so that the lubricants cannot get into the food, he has no problem except that of possible migration of components of the packaging material.

The package maker, too, should have no problem if the machines he uses are designed, operated and maintained in such a way that the packaging material does not pick up any lubricant. If it does, he still may be all right if it can be shown that the lubricant does not migrate into food.

The builder of food machinery has no direct responsibility for food safety. But, if his machines are not made in such a way as to prevent migration of the lubricant into food, he will not sell very much equipment.

How then can the lubricant manufacturer best serve these customers and at the same time avoid difficulties with the FDA? Machinery has to be lubricated. First of all, he should not sell or recommend certain types of greases or lubricants to food processors in order to prevent the possible undesirable effects of accidental contamination. This is a good practice even though he has no control over how the product is used once it has been sold. Second, he should not sell or recommend a lubricating oil or grease to a food processor or packager where, because of the conditions of operation or the design of the machinery, some of the lubricant is continually getting into the food. In these circumstances the lubricant becomes an additive under the law and no conventional lubricating oil or grease has yet been approved or granted an extension. The same practice seems wise if the customer makes food packaging materials unless it can be definitely shown that none of the lubricant which might be picked up by the packaging material migrates into food from the packaging material.

A lubricant manufacturer should not be expected to do all the tests required by the FDA to establish the safety of his oils and greases or to permit the establishment of a residue tolerance. He sells a variety of such products. At the risk of seeming repetitious, he has no control over their use once they have been sold. He cannot be expected to know all the possible uses or misuses of his products once they leave his plant.

What happens after March 5, 1961 when all extensions terminate? The law has no provision for any extensions beyond that date and the FDA has no plans to ask Congress to amend the law. The point is academic as far as lubricants are concerned because no conventional lubricants have been granted extensions. And while extensions may still be granted for materials in use before January 1, 1958, they would also terminate on March 5, 1961. The four months intervening are not enough to make all the necessary tests to establish the safety of a lubricant as a food additive if such studies are started now.

A word about guarantees. It was said earlier that about a year ago many customers were asking if the lubricants they purchased were "cleared" by the FDA. A little later lubricant makers were being asked to sign guarantees that their products would produce no food additives problem when used in food products. The Amendment does provide that anyone charged with a violation of the law may be freed of the responsibility if he can show that the material in violation was delivered to him in good faith under a valid guaranty. Mr. J. Kenneth Kirk, assistant to the Food and Drug Commissioner, had this to say about guarantees at a meeting of the Chemical Specialties Manufacturers Association on May 18, ". . . a food manufacturer who obtains from the supplier . . . a guaranty that the use of his products as directed would result in no contamination of the food, would not be relieved of the responsibility if it turned out that the food did actually become contaminated, although, with or without a guaranty, the question might well arise as to whether the . . . supplier should be held responsible for having caused the violation involving the finished food." He said further that "The FDA has advised inquirers that the guaranty of an ingredient or of a component used in the plant which is not the same as the finished product shipped from the plant would have no legal standing but that it would seem that any manufacturer would be willing to guarantee to a user that he is delivering precisely what he represents his product to be."

To Summarize

1. Lubricant manufacturers have no direct responsibility for the safety of a food. Their products are sold as lubricants and are not intended to become food components.

2. A lubricant may become a food additive if conditions in a food plant are such that the lubricant continually gets into the food product.

3. Lubricant manufacturers can best serve their customers and, at the same time, protect themselves by not selling or recommending their lubricating oils and greases for use where conditions of operation permit continual contamination of food. They should also not sell certain types of lubricants to food plants in order to avoid possible undesirable effects of accidental contamination. ■

About the Author

A. E. Dooley *was graduated from Bethany college in West Virginia. He served as assistant director of the Pennsylvania State Bureau of Industrial Hygiene. From* 1942 *to* 1945, *he was a major in the Sanitary Corps, U. S. Army, attached to the Army industrial hygiene laboratory. He has been an industrial hygienist for Texaco Inc. since* 1947. *He is a member of the American Industrial Hygiene Assn., the American Public Health Assn., and the API medical advisory committee. Mr. Dooley is also chairman of the API interdivisional committee on labeling.*

A CO-OP FIRST—
RAPESEED OIL GREASE

By: M. A. McNichol
Federated Co-operatives Limited

A UNIQUE promotional program on a new product was launched by Federated Co-operatives Limited when it began marketing a grease containing rapeseed oil in March, 1960.

Federated Co-operatives Limited, with its head office in Saskatoon, is the central wholesale and procurement agency owned by nearly 500 consumer co-operatives in Saskatchewan, Manitoba and northwestern Ontario. Among its facilities for servicing its members are a co-op refinery at Regina, Saskatchewan; a coal mine at Drumheller, Alberta; lumber mills at Canoe and Chase, British Columbia; a feed mill at Saskatoon and Winnipeg; warehouse and office facilities at Saskatoon, Regina and Winnipeg; and warehouse facilities at Tisdale, Swift Current and Yorkton, Saskatchewan.

The Saskatchewan Research Council, located on the campus of the University of Saskatchewan, was set up to undertake research, both pure and applied in the sciences, which would affect the economy of the province; and rapeseed oil was one of the products that had shown outstanding potential.

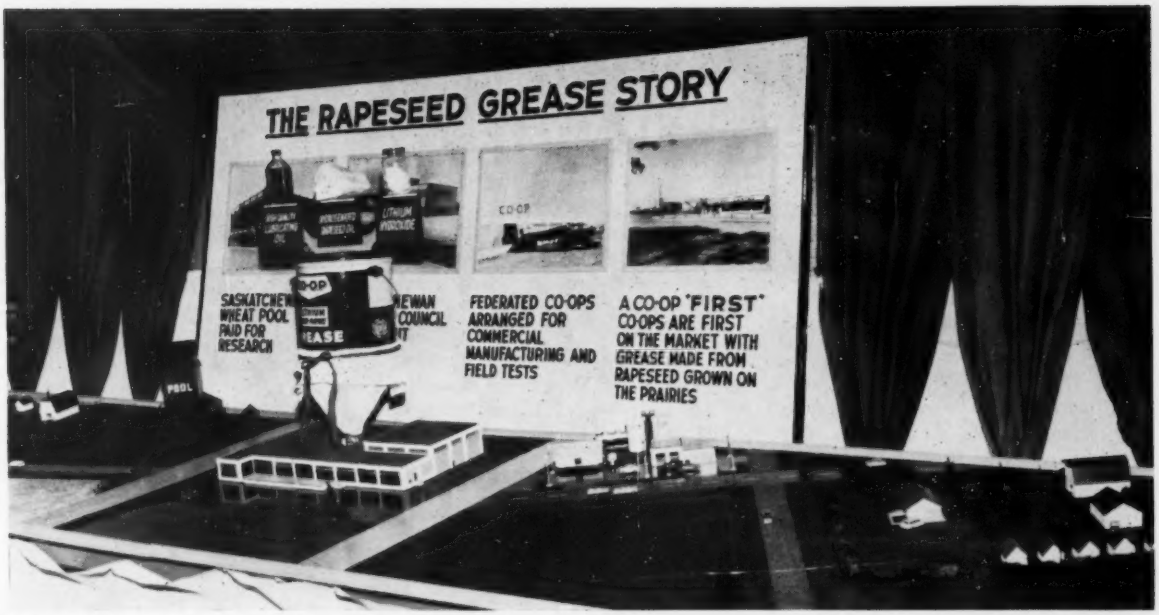
MIKE Nicholaichuck, fuels and lubricants engineer of Federated Co-operatives Limited, and I. S. Evans of the Saskatchewan Research Council, check test results of the new Co-op lithium multi-purpose grease as shown on a recording spectrophotometer.

The Saskatchewan Wheat Pool, a producer marketing co-operative, initiated the research work on the rapeseed oil by a grant to the Saskatchewan Research Council. Mr. I. S. Evans of the Saskatchewan Research Council headed a group of research workers on this project.

Basing their experiments on a suggestion from the Federated Co-operatives Limited, the initial research into the possible use of rapeseed oil in the manufacture of a satisfactory multi-purpose grease was conducted by this research council. This council also exposed their work to a number of grease manufacturers to corroborate their laboratory experiments with more practical pilot plant runs.

Mr. Mike Nicholaichuck, fuels and lubricants engineer with Federated Co-operatives Limited, realizing the sales potential of a grease using a farm-grown product, approached a number of grease suppliers in an effort to develop a commercial lithium multi-purpose grease using rapeseed oil in place of imported fatty acid materials. In August, 1959, 5,000 pounds of a mixed calcium and lithium rapeseed soap-base grease were produced and extensive field test work was carried out. The grease proved very popular. Among the comments noted were the ease of handling and the good stability characteristics.

A number of Canadian and American grease suppliers also conducted research work towards developing rapeseed grease which could be marketed commercially by Federated Co-operatives Limited. One supplier, who had been following the work of the research council very closely, successfully manufactured a commercial batch of this rapeseed multi-purpose grease, using as a portion of the base fatty acid mate-


THE RAPESEED GREASE STORY
SASKATCHEWAN WHEAT POOL PAID FOR RESEARCH
FEDERATED CO-OPS ARRANGED FOR COMMERCIAL MANUFACTURING AND FIELD TESTS
A CO-OP "FIRST" CO-OPS ARE FIRST ON THE MARKET WITH GREASE MADE FROM RAPESEED GROWN ON THE PRAIRIES

A PORTION of the Federated Co-operatives Limited display which was shown at the Silver Anniversary celebration of the Co-op Refinery at Regina. The display indicated how the farm-grown rapeseed is sold through a pool elevator, then made into grease which is distributed by Federated Co-operatives Limited to the consumer co-ops and finally purchased by the farmer again.

rial, Canadian hydrogenated rapeseed oil. This finished lithium grease exhibited outstanding characteristics regarding work stability, resistance to bleeding and oxidation, in addition to a remarkably good appearance. The grease using Canadian hydrogenated rapeseed oil, in combination with other fatty acids, compares favorably with multi-purpose greases marketed.

Lithium multi-purpose grease with rapeseed oil was first introduced at a conference of some 350 retail co-operative managers held in Winnipeg during March, 1960. The slogan "A First For You—Mr. Farmer—Lithium Multi-Purpose Grease With Home-Grown Rapeseed Oil" was adopted and used as a theme in the promotional and advertising material.

As part of the promotional program for this grease, a 4-ft. x 16-ft. exhibit was prepared, illustrating the theme with scale models of a farm, a Saskatchewan Wheat Pool elevator, Federated Co-operatives Limited and a retail co-op. The exhibit was intended to emphasize that especially in times of surpluses of farm products, the farmer could help himself by purchasing this grease.

The exhibit was first displayed at the 25th Anniversary celebration of the Co-op Refinery, held in Regina on June 18, 1960. Over 40,000 people viewed the exhibit. It also attracted considerable attention at both the Saskatoon and Regina exhibitions.

Sales of this new grease in the first three months have been in excess of 100,000 pounds. During a time when farm grease sales have been declining, Federated Co-operatives Limited has been able to show a substantial increase in grease sales.

The complete effects of this novel grease promotional program on the sales of other petroleum products will not be known for another year, but results to date indicate favorable acceptance of the product. ■

About the Author

M. A. McNichol *obtained his BS degree from the College of Engineering, University of Saskatchewan, Saskatoon, Sask., in* 1949. *He joined the staff of Federated Co-operatives Limited in* 1955 *as a fuels and lubricants engineer. At the present time, he is manager of product and supply with the company. He is a member of the Association of Professional Engineers of Saskatchewan and the Engineering Institute of Canada.*

Literature and Patent Abstracts

Composition

High Melting Point Lubricating Greases

A composition and method are described by Pattenden, Sproule and Norton, in U.S. Patent 2,937,-993 (assigned to Esso Research and Engineering Co.) whereby lithium or sodium salts of dicarboxylic acids are used as thickeners in lubricating greases. The particular feature of such lubricants is dropping points above 500°F. Such thickeners are prepared by the reaction of the metal base with either a dibasic acid ester or with a mixture of an aliphatic dicarboxylic acid and a monohydric aliphatic alcohol. Further, a mixture of a higher fatty acid, such as stearic, with the diester can be used in forming the thickener.

Preferred dibasic acids are adipic and sebacic which may be used in conjunction with 2-ethylhexanol or as diesters of the same. The thickener formation may take place in the presence of a portion of the lubricating fluid which may be mineral oil, silicone fluid or even a diester. In the latter case, only a portion of the diester will react with the alkali.

For example, a lubricating grease was prepared by mixing 15 grams of di-(2-ethyl hexanol) sebacate with 81.7 grams of DC 710 silicone fluid, adding 3.3 grams of lithium hydroxide monohydrate and heating to 400°F for about 15 minutes. At this temperature the 2-ethyl hexanol formed during the reaction was driven off almost as rapidly as it formed. It is essential that the alcohol be entirely driven off, otherwise a soft product will result. Upon cooling, the above product had a micropenetration of 80 and a dropping point above 500°F.

Silica Thickened Lubricating Grease

Milberger in U.S. Patent 2,945,-809 (assigned to the Standard Oil Co., Ohio) describes a lubricating grease consisting of a lubricating oil thickened with 7 to 12 per cent of a silica aerogel. In order to produce lubricants which have temperature stability in the range of 200 to 400° F, the silica should be prepared from a hydrogel containing 3 to 8 per cent SiO_2 by converting the hydrogel to an alcogel and converting the alcohol to a gas without destroying the gel structure of the silica. If properly prepared, the silica will then have an average secondary agglomerate particle size before mixing with oil of 1 to 6 microns. The average particle size after simple mixing with oil will be not over 0.25 micron. With such a silica, lubricating greases can be prepared without milling.

Thus, at a temperature of 85° F, 7 per cent of the preferred silica was slowly added to a solvent refined oil of 250 SUS at 210°F. Immediately after the last of the silica had been wetted, the mass was subjected to 1000 strokes in a grease worker. The product then had a penetration of 296. A portion of this lubricant was heated undisturbed to 400°F, cooled to room temperature and stirred vigorously. The original sample had a micro-

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penetration of 99 which changed to 153 after the heating and cooling cycle.

Additives, such as Amine "O," can be added to these lubricating greases to improve the resistance to water.

Copper Corrosion Resistant Lubricating Greases

According to Dreher and Carter (U.S. Patent 2,945,813, assigned to California Research Corp.) lubricating greases in which the thickener is a metal salt of a terephthalamic acid are inhibited against corrosion of copper if 0.2 to 15 weight per cent of a metal soap of a hydroxy-substituted fatty acid is included in the composition. While metals selected from the group consisting of alkali metals and alkaline earth metals can be used, the preferred compounds are those of sodium. Also the preferred hydroxy-substituted fatty acid is 12-hydroxystearic acid.

For example, a mixture of 41.6 pounds of methyl N-"octadecyl" terephthalamate, 10.4 pounds of 12-hydroxystearic acid, and 84 pounds of a California solvent-refined paraffin base white oil having a viscosity of 100 SUS at 100°F was heated to 300°F until a uniform solution was obtained. The mixture was cooled to 140°F, and 5.8 pounds of sodium hydroxide (in 50 per cent aqueous solution) were added. The mass was then heated to 250°F where 6 pounds of an oxidation inhibitor consisting of two-thirds trimethyl dihydroquinoline polymer and one-third phenyl alpha-naphthylamine were added. The mixture was then heated to 310°F and 252.2 pounds of diisooctyl azelate added before further heating to 380°F, milling and cooling.

The finished lubricating grease had a worked penetration of 271 and passed a copper strip corrosion test when held in a bomb under oxygen pressure for 20 hours at 210°F. Two similar lubricants, one of which contained some sodium stearate but both of which had no

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sodium soap of 12-hydroxystearic acid present, failed to pass the copper strip corrosion test.

Open Gear Lubricants Containing Silicones

Sproule and Rogers (U.S. Patent 2,946,749, assigned to Esso Research and Engineering Co.) found that the addition of 0.5 to 1.5 per cent of certain silicone fluids to asphaltic base open gear lubricants increased the lubricating life of the compounds. Dimethyl silicone polymers having a viscosity of 300 to 3,000 SUS at 210°F and thickened with 4 to 20 per cent of fine silica are the preferred additives.

The lubricating life of these viscous gear compositions was determined by applying the lubricant to open gears operating at a temperature of 100°F, carrying a load of 260 pounds per tooth and operating at a peripheral speed of 134 feet per minute. Provision was made to drip water on the gears at a given rate if desired. The lubricant life was arbitrarily chosen as the point when a ½-inch bare area on the bearing surface was visible when viewed under ultra-violet light.

An asphaltic gear lubricant having a viscosity of about 2,000 SUS at 210°F and a softening point of about 100°F was used in tests. With no water contact, the lubricant life was 72 hours. This was increased to 186 hours after the addition of 0.1 per cent of a mixture of 90 per cent silicone fluid and 10 per cent fine silica. With a water contact rate of 15 cc per minute, the life of the base lubricant was 23 hours, which was increased to 50 hours after adding 0.1 per cent of the silicone composition.

Liquid Hydrocarbon Gels

While the compositions claimed by Steiner and Gibson in U.S. Patent 2,946,748 (assigned to Swift & Co.) are proposed for gelling hydraulic fluids, the information may be of value to producers of lubricating greases.

The gelling agents commonly used in the above fluids consist of soaps of fatty acids having 14 to 18 carbon atoms in their chains. It is claimed that the presence of as little as 10 per cent of fatty acid soaps of a carbon chain length of 20 or more substantially increases gel viscosity and decreases water sensitivity of the gel. Hydrogenated fish oils are desirable for furnishing the longer chain fatty acids. The preferred soap is formed by using a 50/50 mixture of ammonium hydroxide and caustic soda.

Pigment Thickened Lubricating Greases

High temperature lubricating greases having EP qualities are described by Odell and Lyons in U.S. Patent 2,946,750, assigned to Texaco Inc. The thickeners for such lubricants are the reaction products of arylamine compounds with heteropoly acids of the class of phosphomolybdic, phosphotungstic and phosphotungstomolybdic acids. Such classes of dyes are known commercially as PMA, PTA and PTMA pigments.

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NLGI-11

Thus a product was obtained by precipitating methyl violet B with phosphomolybdic acid in acid solution. Thirty per cent of this pigment was mixed with a naphthene base oil having a viscosity of 310 SUS at 100°F and given two passes through a Premier Colloid mill at 0.002 inch clearance. A smooth NLGI No. 2 grade of lubricating grease resulted with a dropping point above 500°F. Tested on a 4-ball apparatus, the seizure load was 52 kg. In comparison, the same oil thickened with silica had a seizure load of 29 kg. Other lubricating fluids can be thickened with such pigments and additives can also be used in the lubricants.

Wire Rope Lubricant

While the composition described by Vierk and Karr in U.S. Patent 2,947,697 (assigned to Sinclair Refining Co.) is suggested for use as a lubricant for wire ropes, similar mixtures may well find application as gear compounds or even lubricating greases.

It was found that by including 3 to 6 per cent of polyethylene in mixtures of blown asphalt and lubricating oil, the softening point was raised and the flexibility at low temperatures was improved.

Thus, a mixture of 64 per cent air blown asphalt and 36 per cent of a solvent refined Mid-Continent oil having a viscosity of 155 SUS at 100°F had a softening point of 106° F. A wire rope coated with this mixture, cooled to -5°F and flexed, showed a few tiny cracks in the coating. A similar mixture, to which was added 5 parts of a polyethylene having a molecular weight of 2,000, had a softening point of 142°F and when used as a wire rope coating developed no cracks upon flexing at -5°F.

EP Lubricating Grease

According to British Patent 837,-712, issued to C. C. Wakefield & Co. Ltd. (George H. Thornley, inventor), addition of one to five per cent of the gamma isomer of benzene hexachloride confers EP qualities to the product. Thus, a product consisting of 88.5 per cent mineral oil, 10.5 per cent lithium stearate and 1 per cent gamma benzene hexachloride had good load-carrying capacity. No test data were given.

Composition and Processing

Silicone Fluids Thickened with Lithium Soap

In Japanese Patent 1004, dated February 28, 1859, Nagai and Sakurai as inventors but Shin-Etsu Chemical Industry Co., Ltd., as claimant, describe the manufacture of a lubricating grease consisting of silicone fluid thickened with a lithium soap.

Thus, a mixture of 64 per cent air petroleum naphtha boiling between 160 and 180°C and 20 grams of 9,10-dihydroxystearic acid was heated to 80 to 90°C. To this mixture were added 50 ml. of a aqueous solution containing 5 grams of lithium hydroxide. After heating this mass to 130 to 140°C, 200 grams of polymethylsiloxane were added gradually. Finally the solvent was evaporated to give a lubricating grease having a dropping point of 193°F.

Process

Continuous Grease Manufacture Using Coated Alkali Metal Saponifying Agent

Alkali metal hydroxides and particularly lithium hydroxide are made free-flowing so that they can be fed by means of a vibratory meter to continuous grease manufacturing equipment. For the purpose the lithium hydroxide is coated with a dry, hydrophobic (water hating) powder which is preferably hydrated lime. This suggestion is made by Beerbower and Meyer in U.S. Patent 2,946,751, assigned to Esso Research and Engineering Co. It is important that the coating material be acceptable in the final lubricating grease and, when this coating consists of hydrated lime, the

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Representative—S. A. Bennett

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Representative—Theodore Humphrey

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Representative—Warren T. Trask

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The Farval Division Eaton Manufacturing Co.
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Valvoline Oil Company
Division of Ashland Oil & Refining Co.
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Representative—R. L. Sailer

SUPPLIERS OF EQUIPMENT FOR MANUFACTURING LUBRICATING GREASES

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Lithium Corporation of America, Inc.
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Swift & Company
165th & Indianapolis Blvd., Hammond, Ind.
Representative—F. H. Beneker

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Witco Chemical Company
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TECHNICAL AND RESEARCH ORGANIZATIONS

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Representative—S. L. Cosgrove

The Chek-Chart Corporation
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Chemico (Pty.) Ltd.
Miller and 7th Streets, P. O. Box 6349,
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Representative—O. Richter

Compagnie Francaise de Raffinage Direction Recherches et Procedes
22, Rue Boileau, Paris (16e) France
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CMR—Courtel, 4 Place Bir Hackeim
Rueil—Malmaison (S. et Oise) France

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Representative—R. Gillerot

Phoenix Chemical Laboratory, Inc.
3953 Shakespeare Ave., Chicago 47, Ill.
Representative—Mrs. G. A. Krawetz

finished product be a mixed base lithium-calcium soap thickened material.

The coated alkali metal base is most conveniently made into a slurry with a portion of the lubricating oil and this slurry, in turn, is fed to the continuous manufacturing equipment. In case there is a tendency for the base to settle out of the slurry, a small proportion of fatty acids or better still 3 to 15 weight per cent of the finished lubricating grease can be included in the slurry.

For example, an alkali-oil slurry was prepared by using the following weight per cent of materials: lithium hydroxide monohydrate 23.67, hydrated lime of 200 mesh 13.33, lithium-calcium base lubricating grease 5.00, and oil of 70 SUS at 210° 58.00. First, the lithium hydroxide and the lime were mixed in a Patterson Kelly Blender for 15 minutes. This mixed powder was then metered on an Omega Machine Feeder to a Stratco Contactor with the oil and the lubricating grease. This gave a slurry which showed no tendency to separate in one month at 80°F. This alkali slurry was metered by a screw type pump at 6.9 pounds per minute, 200°F, and 100 psi, while animal fatty acids were metered by a gear pump at 15.3 pounds per minute, 180°F and 100 psi. The streams joined in a Lancaster Disperser driven at 3500 rpm. The soap mixture, then at 260°F, was led through a scraped surface heater, leaving at 296°F. Oil at a rate of 77.8 pounds per minute, 135°F and 100 psi was metered in and mixed with the soap in a second disperser. The resulting lubricating grease at 235°F was passed through a pressure reducing valve and dehydrated in a Cornell Homogenizer at 21 inch vacuum, cooled to 135°F and milled in a Charlotte Colloid Mill at .015 inch clearance.

The Literature and Patent Abstracts column is written for NLGI by C. J. Boner, director of laboratories for Battenfeld Grease and Oil of Kansas City, Missouri.

The finished product containing 12.5 per cent soap, one-third of which was calcium and two-thirds lithium, was smooth in texture. It had a worked penetration of 270 and passed the ASTM Wheel Bearing Test.

Equipment

Flow Sheet and Short Description of Grease Manufacturing Process

The Petroleum Engineer for Management, April 1960 p. 19-20.

A flow sheet and short description of the Stratco grease manufacturing equipment is given. This is published in a form where it can be inserted in a notebook. The major equipment consists of an oil circulation heater, a contactor, a scraper kettle and a grease polisher. Heats up to 600°F are claimed for the system and time cycles of 1½ to 2 hours for calcium base lubricating greases and 2 to 3 hours for sodium base lubricating greases.

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People in the Industry

Executive Changes at Battenfeld (Kansas City)

A number of executive changes were announced by Battenfeld Grease and Oil corporation of Kansas City, effective last December 1. Hugh H. Bruner was elected president and chief executive officer of the company, replacing A. J. Daniel, who will continue to serve on Battenfeld's board of directors

H. H. Bruner

and plans to remain active in association affairs for the firm. Charles E. Gore was elected vice-president of lubricant sales, and Donald H. Vance was elected treasurer and a member of the board of directors.

Bruner is the third president of the 45-year-old corporation, which is well known in the petroleum industry as a lubricants manufacturer and in the building industry as a manufacturer of building and maintenance products. The late J. R. Battenfeld, founder and first president of the firm, was also one of the founders of NLGI and served as the Institute's first president in 1933-34.

A graduate of the University of Kansas, Bruner is a combat veteran of the European theatre in World War II, where he attained the rank of captain and was awarded the Silver Star with cluster, the Purple Heart, and the Croix de Guerre with palm. He became associated with Battenfeld in the position of assistant treasurer and comptroller in 1948, after experience with Arthur Anderson and company, certified public accountants. In 1952 he was elected executive vice-president of Battenfeld.

Long active in NLGI financial affairs through his close association with A. J. Daniel, NLGI's nine-term treasurer, Bruner established many of the business practices and procedures of the Institute's national office during a reorganization in early 1956. He has assisted the treasurer in preliminary planning on NLGI budgets and, for the past eight years, has been convention auditor of the NLGI annual meetings.

C. E. "Chuck" Gore joined Battenfeld in 1953, serving as a territory representative. He was named sales manager of the lubricants division in 1955 and served in that capacity until his recent election to vice-president. Don Vance became office manager of the company in 1955, the year he joined the firm. Last year he was promoted to assistant treasurer.

A. J. Daniel will continue in his position of treasurer of NLGI. He is a past president of the Institute and has been Battenfeld's Company representative since 1945.

Cubicciotti Named Witco Vice-President

The promotion of Rudolph Cubicciotti to administrative vice-president of Witco Chemical company, Inc., was announced last month.

In his new post, Mr. Cubicciotti will direct the planning and co-ordination of all activities that relate directly to future corporate growth. His responsibilities in this area extend to all Witco divisions, including Sonneborn Chemical and Refining corporation, Ultra Chemical Works, Inc., and Witco's tar products and Pioneer Products divisions.

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Prior to his promotion, he was vice-president of Sonneborn Chemical and Refining corporation — a wholly-owned Witco subsidiary. He joined Sonneborn in 1943 and, before that, was product sales manager for Union Oil company of California. He is a graduate of the University of California with a B.S. in Chemical Engineering.

A long-time member of the American Petroleum Institute, Mr. Cubicciotti served as the 1954 chairman of its Lubrication Committee and is presently chairman of its Motor Oil Study Panel. He is also a director of the National Lubricating Grease Institute, of which he was president in 1958.

He is active in the affairs of several yachting and boating organizations and is presently vice-commodore of the Eastern Cruiser association.

Witco manufactures and markets a broad line of chemicals and allied products including detergents and detergent chemicals, phthalic anhydride, white oils, solvents, carbon black, resins for urethane foams, petroleum sulfonates, petroleum waxes, lubricating oils, metallic soaps, tar and asphalt products, and building product specialties.

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Nelson Morris Elected President of Darling

Nelson Morris II has been elected president of Darling & Company, one of the country's largest independent rendering firms. He succeeds William H. McCausland, who has become chairman of the board.

The position of board chairman has been vacant since the death of Charles A. Alling last April. Mr. McCausland, associated with the company since 1910, served as president since December, 1954.

Mr. Morris joined the firm in 1946. He was general manager of the Buffalo, N. Y., plant from 1952 to 1954 and then returned to Darling's Chicago headquarters as assistant to the vice-president in charge of sales. He has been vice-president in charge of sales for the past four years.

In another management promotion, Ralph B. Dainty has been named vice-president in charge of raw material. He joined the company in 1939 and returned following military service during World War II.

Darling & Company, established in 1882, manufactures fertilizers, animal feeds, fatty acids, glycerine, glue, tallows, greases, and other animal by-products. In addition to its general offices and plants at Union stock yards in Chicago, the company has plant facilities at Cleveland and Cincinnati, Buffalo, Detroit, East St. Louis and Cairo, Ill.; Cedar Rapids and Alpha, Iowa; Webb City, Mo.; and Chatham, Ontario.

Japanese Member Adds Two to Staff

Two new staff members have joined Kyodo Yushi Co., Ltd., NLGI member in Tokyo, Japan. Kunihiko Kofune and Akira Kofune began work with this manufacturing company immediately after graduation this year, and have been learning the way of the business of warehousing, transportation and accounting.

Kunihiko Kofune is the son of S. Kofune, president of Kyodo Yushi. He is a graduate of Aoyama Gakuin university's department of economics. Akira Kofune is a nephew of S. Kofune and son of the managing director of the company in charge of sales. He is a graduate of Waseda university's department of commercial science.

Both young men hope to visit lubricating grease establishments in the United States in the future.

Sun Oil Appoints Two General Sales Managers

Sun Oil Company has announced the appointment of two general sales managers—Elmer R. Bradley and Marcus A. Markley.

Thomas S. Horrocks, marketing vice-president, said Mr. Bradley will be in charge of the merchandising and sale of motor products, including automotive fuels and lubricants and tires, batteries and accessories, both directly and through wholesale distributors and commission agents. He also will direct distribution operations.

Mr. Markley will be in charge of industrial, wholesale, fuel oil, and foreign sales, and the sale of liquefied petroleum gas.

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Industry News

Seeks New Uses for Lubricating Greases

Ever aware of the need for new markets, S. C. M. Ambler, product line coordinator for British American Oil company in Toronto, Ontario, Canada, recently polled B-A employees via the company magazine, B-A *Commentator*. Under the heading, "Wanted, New Ideas for Greases," the entire organization was solicited.

Ambler found that when given the opportunity and not hampered by preconceived ideas, people will come up with suggestions of value. Some of the non-lubricating uses for grease he received:

Protecting sore spots on farm animals . . . coating the bottom of fence posts and telegraph poles . . . coating ledges on buildings to prevent birds roosting . . . coating spray booth rear panels and extraction fans for easy removal of accumulated paint . . . hand cleaner and skin conditioner for mechanics . . . prevents cow's udders from chapping in the winter . . . rust protector in humid atmospheres.

As a member of the Institute's Board of Directors, concerned with new markets for the industry, Ambler suggested that if all member companies who have internal publications would do the same thing and forward any replies received to NLGI's national office, the letters received will be acknowledged and those of practical worth published in the NLGI SPOKESMAN.

ADM to Build New Research Center

Archer-Daniels-Midland company, from whose laboratories have come hundreds of new chemicals, plastics, protective coatings, foods and other products, has announced that it will construct a new research center to broaden even further its research activities.

John H. Daniels, ADM president, said the new science facility will be located on a 73-acre tract overlooking the Minnesota river valley in Bloomington, Minn., a suburb of Minneapolis.

The research center will bring together the company's research,

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"The new facilities will enable ADM to expand its industrial research," Daniels said. "We will have added space for research in our present fields as well as for research in new directions that we contemplate in the future."

The ADM president said ground will be broken for the research center in mid-June, 1961, with completion scheduled for 1962.

Plans to Establish Qualified Products List For Hydraulic Fluid, Etc.

The Ordnance Corps, Department of the Army, has announced the intention to establish a Qualified Products List for Hydraulic Fluid, Petroleum Base, For Machine Tools, under Specification MIL-H-46001 (ORD). Companies which have a product meeting the requirements of this specification are urged to contact the Commanding Officer, Rock Island Arsenal, Rock Island, Ill., Attention: Laboratory, for an opportunity to have their products tested, since in making future awards, awards will be made only for such products as have been tested and approved for inclusion in the Qualified Products List.

Research on High-Speed Rotary-Shaft Face Seals Begun by Battelle

A major research program to provide a more basic understanding of factors involved in the design and use of high-speed rotary-shaft face seals has begun at Battelle Memorial Institute. The program is being jointly sponsored by "more than a dozen large American manufacturing companies interested in advancing shaft-seal technology," according to C. M. Allen, specialist in bearings, seals, and lubrication technology at the Columbus, Ohio, research center.

In explaining the new program, the Battelle technologist pointed out that the leakage of high-speed mechanical shaft seals has posed a serious problem in the design and operation of advanced high-speed turbomachinery. "Shaft seals of 50,000 to 100,000 revolutions per minute and higher are now designed into modern turbocompressors, as for example, in the accessory equipment for commercial jet aircraft. We can expect high-speed seal-design and leakage problems to become critical as the use of high-speed turboequipment becomes more universally employed. Our own research experience in this area and the number of inquiries we have received suggest strongly that, in the absence of a well developed technology, many seal problems have had to be solved by expensive cut-and-try methods that contribute little to the general improvement of seals."

Presently on a one-year schedule, the program will be directed toward the dynamic behavior of high-speed liquid-lubricated face seals. Basic experimental data will be obtained on the performance of such seals, with special emphasis on the behavior of the primary seal elements. By carefully planning and conducting these seal experiments, it will be possible to control and monitor the behavior of the entire seal system and its supporting structure. Analysis of data obtained in these experiments will make it possible to evaluate the importance of the various parameters involved. The program is expected to provide seal-design data of direct value in the design, installation, and application of high-speed rotary-shaft face seals and machinery.

Principal investigators for the program are Frederick K. Orcutt and J. Clarence Bell.

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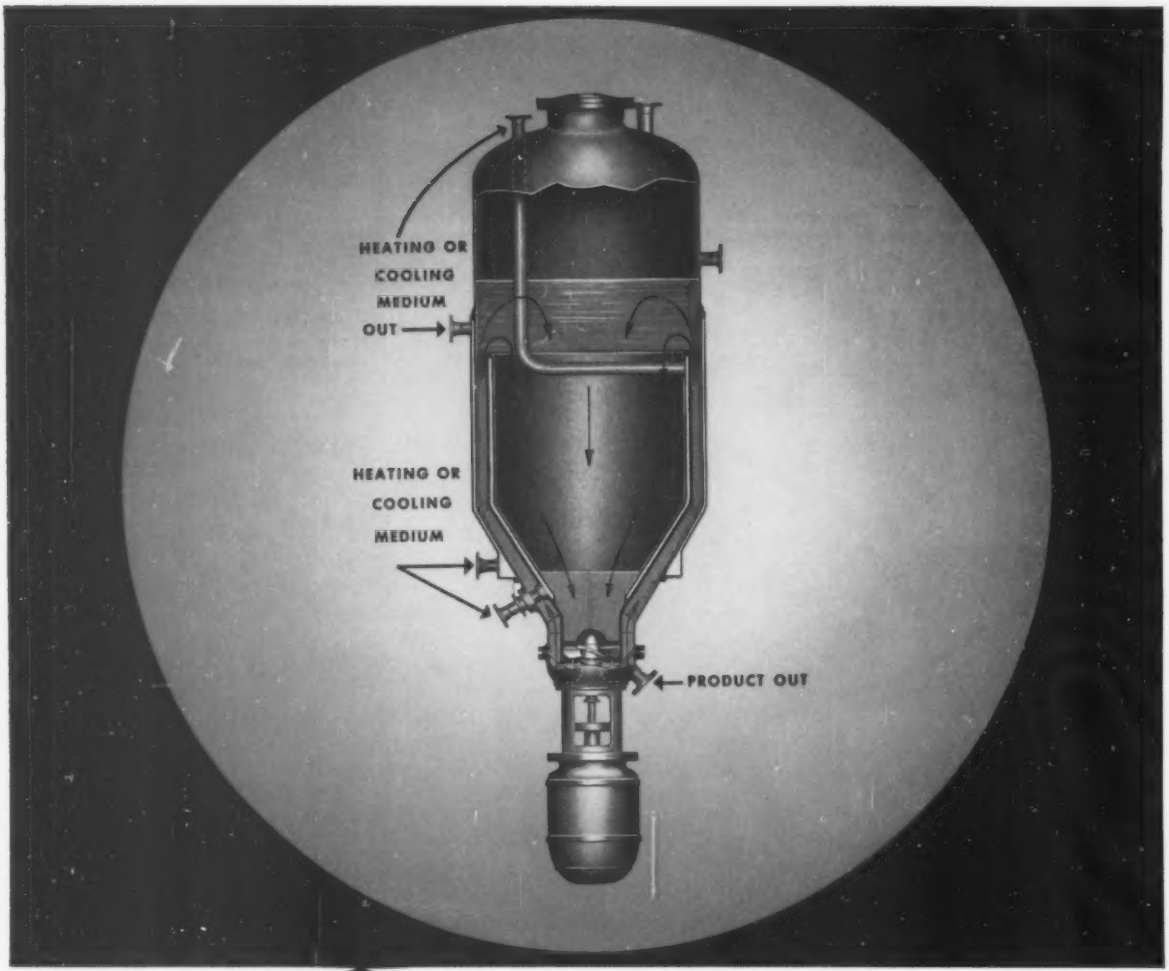

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